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MECHANICAL BEHAVIOR OF A SiC-FIBER/Si_3N_4 COMPOSITE

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September 1990

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U.S. ARMY MATERIALS TECHNOLOGY LABORATORY
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Technical Note by

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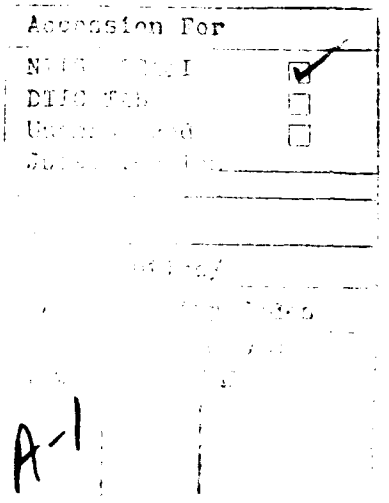
ABSTRACT

The room and elevated temperature mechanical properties of a silicon carbide fiber/silicon nitride matrix composite were examined. The findings showed that the addition of fiber layers to a ceramic matrix eliminates the catastrophic brittle failure at room temperature. As the test temperature increases, the tendency for failure to proceed in a brittle manner increases. This is most likely due to an increase in the fiber/matrix bond strength which does not allow for fiber pullout and crack bridging to occur.



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INTRODUCTION

Over the past 20+ years tremendous strides have been made to improve the strength of advance ceramic materials for structural applications. However, the same type of progress has yet to be made to improve the toughness of these monolithic ceramics materials. As a consequence, ceramists and processing engineers have turned to the development of a relatively new class of materials: ceramic matrix composites (CMC). In this class of materials the toughness is increased through the addition of a second-phase material; i.e., particulates, whiskers, or continuous fibers. The additional phase(s) can increase toughness by one or more of the following mechanisms: crack deflection, crack branching, crack bridging, microcracking, pullout of the second phase, or absorption of the crack tip energy.

In this report we evaluated a continuous SiC fiber-reinforced silicon nitride ceramic. The introduction of layers of fibers to a monolithic ceramic material increases the toughness by deflecting or bridging the crack.[1] In order for toughening to occur by either mechanism, the bond between the fiber and matrix must be controlled; that is, the interfacial shear strength must be optimized so as to deflect the crack along the weak fiber/matrix interface allowing the fiber to slip within the matrix. As the applied stress is increased, debonding will continue until the fiber fractures and is pulled from the matrix. If the bond is too strong the composite will behave like a monolithic ceramic and fail in a brittle manner.

MATERIAL

A silicon nitride matrix, continuous silicon carbide fiber reinforced composite was obtained from Textron Specialty Materials, Lowell, MA. The material was fabricated in July, 1989. The composite is comprised of a silicon nitride matrix with 5 w/o Y_2O_3 and 1.25 w/o MgO additions, and was reinforced by 30 v/o SiC monofilament fibers which were unidirectionally aligned in the billet. The billet size was 100 mm x 125 mm x 13 mm.

The fibers were SCS-6 grade which are processed by chemical vapor deposition (CVD) of β-SiC onto a 33 μm diameter graphite core. An outer layer of carbon, approximately 3 μm thick, is then deposited on top of the β-SiC in order to control interface reactions with the matrix material. The overall fiber diameter is nominally 143 μm.

The SCS-6 fibers were unidirectionally aligned by clamps at the end of the 125 mm dimension, then the silicon nitride was tape cast onto the fibers to form composite green body mats. Stark grade 232 silicon nitride powder was used. The mats were then stacked onto a graphite mold and hot-pressed at 1700°C and 20 MPa pressure for one hour in a nitrogen atmosphere.

No special heat treatment was given in order to crystallize the grain boundary phase in the silicon nitride. An X-ray diffraction pattern from a specimen cut from the fabricated plate revealed the matrix crystalline phase to be α- and β-Si_3N_4 (in a ratio of about 3:2) and probably melilite (Y_2O_3·Si_3N_4). Silicon carbide was not detected since the spot analyzed had no exposed SiC fibers, and most low angle SiC peaks are masked by the Si_3N_4 peaks.

1 MARSHALL, D. B., and EVANS, A. G. *Failure Mechanisms in Ceramic-Fiber/Ceramic Matrix Composites*. J. Am. Ceram. Soc., v. 68, no. 5, 1985, p. 225-231

Only a limited amount of material was available, permitting only a cursory examination of the high temperature mechanical properties. Tensile strength and tensile fatigue results at 1000°C on this material have been reported previously by Holmes, et al.[2]

EXPERIMENTAL PROCEDURE

The billets were subsequently machined into flexure test bars 3 mm x 4 mm x 50 mm for mechanical property characterization. This configuration is certainly not optimal for generating true tensile failures.[3] On the other hand, the flexure test is eminently suitable for qualitative materials assessment purposes.[4,5] The flexure bars were machined such that the fibers were oriented parallel to the long axis of the bar. Slight unevenness in the laid up fiber layers lead to fibers occasionally being exposed on the sides of the bar.

Three bars were subjected to four-point flexure testing in accordance with MIL-STD-1942, using inner and outer load spans of 20 mm and 40 mm, respectively, and a crosshead speed of 0.5 mm/min. The time-dependent strength of the composite was examined using stepped-temperature stress-rupture (STSR) and isothermal stress rupture (SR) tests.

STSR was used to analyze the time-dependent strength between 1000°C and 1400°C. This testing was done following the procedure outlined by Quinn and Katz.[6] This test allows for rapid screening of the materials' time-dependent behavior over a wide range of temperatures while using a small number of specimens. The procedure involves loading a bar onto a four-point-bend fixture that is in a furnace and heating the furnace to 1000°C in ≈2 hours, in air, with no stress applied to the bar. Upon reaching temperature, a predetermined stress is applied and the bar is allowed to soak for up to 24 hours. If the bar survives this step, then the furnace is heated to 1100°C (in ≈10 min.) while under the same applied stress and again allowed to soak for 24 hours. This cycle is repeated for 1200°C and 1300°C, but at 1400°C the bar is allowed to soak for 72 hours. If the bar fractures or excessive creep occurs the power to the furnace is automatically shut off by a microswitch. The time of fracture is denoted on the STSR plot using an arrow, with the applied stress that caused fracture above the arrow. The symbols for the STSR plot are: (←) failure occurred upon application of the stress at 1000°C; (→) survived the full test cycle through 1400°C; (↓) denotes time of failure between application of the stress but before the full cycle is complete.

SR testing was carried out in a similar manner to the STSR except that the specimen was held at only one temperature until failure or 500 hours had elapsed, whichever came first. Specimens which survived SR testing were subjected to room temperature flexure testing to determine the retained properties of the bar.

Optical fractographic analysis was done on all specimens that failed and the scanning electron microscope (SEM) was used on selected fracture surfaces for detailed analysis.

2 HOLMES, J. W., KOTIL, T., and FOULDS, W. *High Temperature Fatigue of SiC Fiber-Reinforced Si_3N_4 Ceramic Composites*. To be published in the proceedings of the American Ceramic Society Symposium on High Temperature Composites, Dayton, OH, June 1989.

3 LEWIS, D., BULIK, C., and SHADWELL, D. *Standardized Testing of Refractory Matrix/Ceramic Composites*. Ceram. Eng. Sci. Proc., v. 6, no. 7-8, 1985, p. 507-523.

4 DAVIDGE, R. W., and DAVIES, J. J. R. *Ceramic Matrix Fibre Composites: Mechanical Testing and Performance*. Int. J. High Tech. Ceram., no. 4, 1988, p. 341-358.

5 PHILLIPS, D. C., and DAVIDGE, R. W. *Test Techniques for the Mechanical Properties of Ceramic Matrix Fibre Composites*. Br. Ceram. Trans., v. 85, 1986, p. 123-130.

6 QUINN, G. D., and KATZ, R. N. *Stepped Temperature Stress Rupture Testing of Silicon Based Ceramics*. Am. Ceram. Soc. Bull., v. 57, no. 11, 1978, p. 1057-1058.

X-ray diffraction was used to determine the phases present in this CMC. Diffraction patterns for routine phase analysis were generated before and after stress rupture tests at 1200°C by scanning between 10° to 55° 2Θ using CuKα radiation. Diffraction was done on either the surface of the flexure bar or on powdered specimens. A limited residual stress analysis investigation was performed at the surface of a flexure specimen prior to stress rupture testing using the X-ray diffraction method and CuKα radiation. The high angle peaks of α-Si_3N_4 (148.1° 2Θ), β-Si_3N_4 (141.8° 2Θ), and β-SiC (133.6° 2Θ) were used for this analysis.

RESULTS AND DISCUSSION

Room Temperature Properties

Failure in this material at room temperature is of a nonbrittle nature as exhibited by the plot of flexure stress versus deflection for room temperature strength testing, shown in Figure 1. In all cases there is an initial linear elastic region followed by a nonlinear increase in the stress. It is this noncatastrophic failure which shows that this material has improved toughness over the monolithic material. The load required to initiate a matrix crack is ≈520 N (≈430 MPa). These results; in particular, the apparent first matrix microcracking and the apparent ultimate tensile strength, are in excellent agreement with the flexure data generated by Textron.

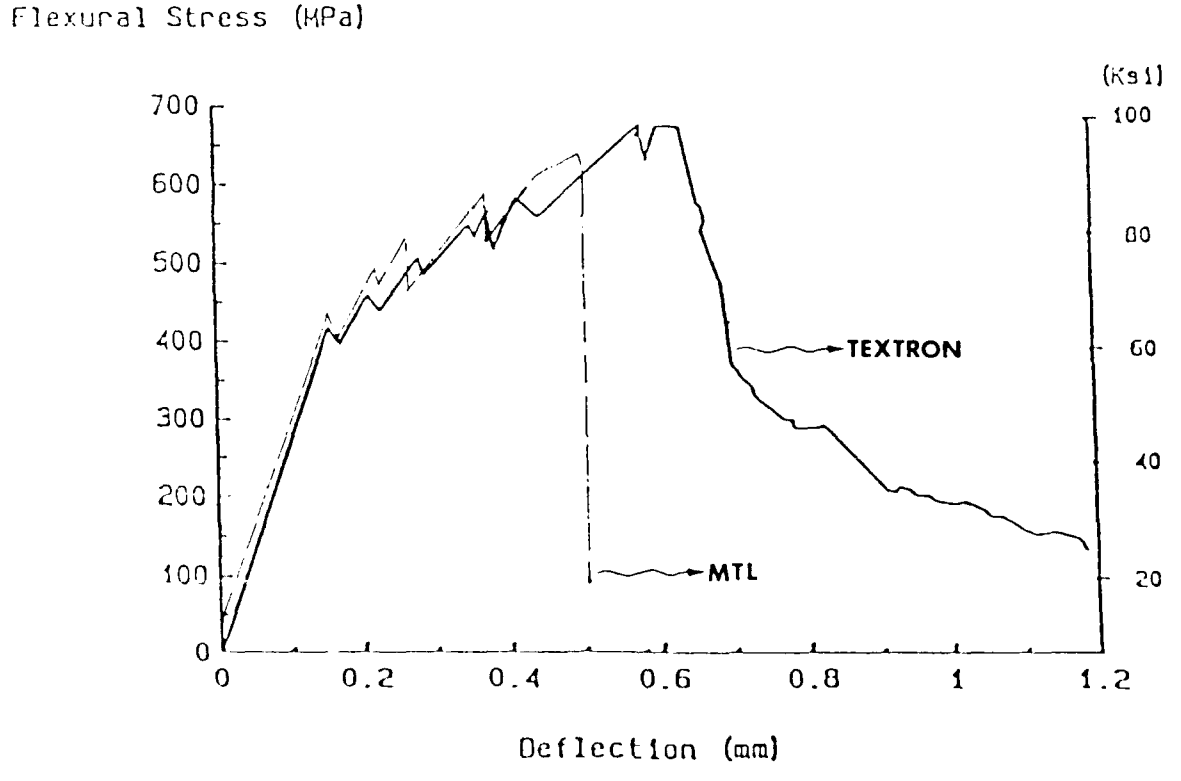
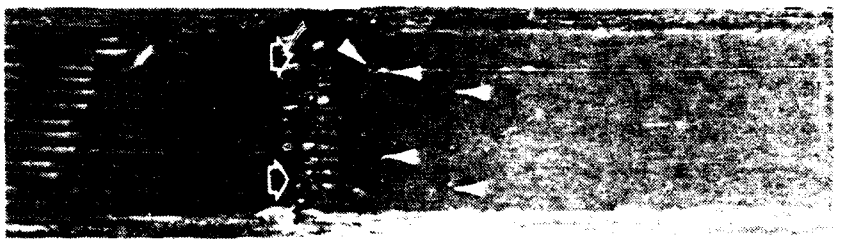


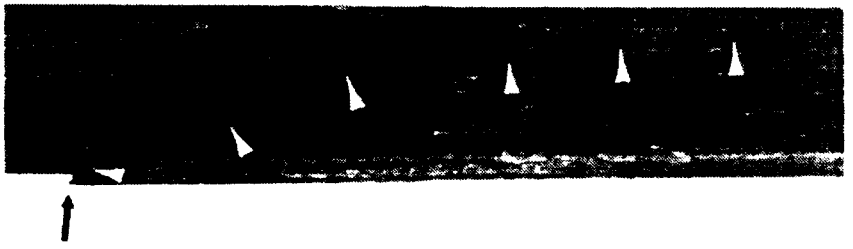
Figure 1. Plot of flexure strength versus deflection comparing the results generated by Textron and this report. The large drop in stress for the bar tested in this report (MTL) is due to the fracture of the bar being interpreted as a failure by the testing machine.

The failure in one of the bars initiated outside the inner gauge length, at a point where the fibers were exposed on the surface, as shown in Figure 2a, and proceeded in a shear-like fashion to the middle of the bar, as shown in Figure 2b. Failure in another bar initiated as a tensile-type failure, but about halfway through the bar the failure mode appeared to shift to a shear-type failure, as shown in Figure 3a. For both of these bars an examination of the tensile surface, as shown in Figure 2a and Figure 3b, showed that there were multiple cracks in the matrix. These cracks were only present between the primary crack and the central section of the tensile surface. The final bar underwent extensive deformation and cracking but did not rupture. It was subsequently broken by hand upon removal from the fixture. Fiber pullout (albeit limited), debonding at the fiber/matrix interface and crack deflect were obvious (see Figure 4). In the latter bar the average fiber pullout length is ≈0.9 mm.



1 mm

Figure 2a. View of the tensile surface of the first room temperature test. Solid arrows show additional matrix microcracks, while open arrows show the main crack.



1 mm

Figure 2b. Side view of the first room temperature test. Black arrow shows the failure origin and the white arrows highlight the crack path

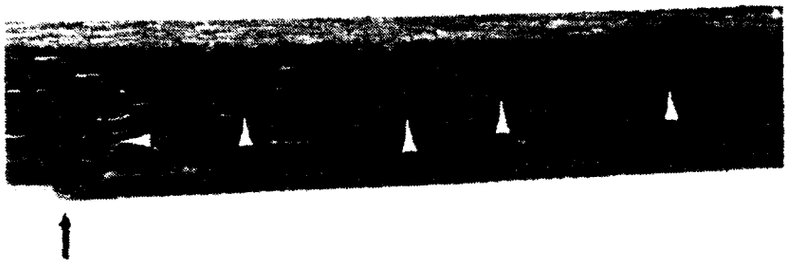


Figure 3a. Side view of the second room temperature test. Black arrow shows the failure origin and the white arrows highlight the crack path

Figure 3b. View of the tensile surface of the second room temperature test. Open arrows indicate primary matrix crack, while solid arrows show secondary matrix microcracking.

Figure 4. Room temperature fracture surface. T denotes the tensile surface, black arrows indicate debonding, and white arrows indicate fiber pullout. Note the aligned layers of SiC fibers (horizontal in the picture) and the coarse size of the fibers.

Examination of the back-reflecting diffraction spectra from the residual stress analysis showed poor peak-to-background ratios as a result of the multiple phases present and the inhomogeneity of the material; i.e., both matrix and fibers within the irradiated volume. Consequently, all residual stress measurements had large statistical errors associated with them thereby reducing the confidence in the calculated stress values.

Stepped-Temperature Stress-Rupture

Figure 5 shows that there is no anomalous behavior in the time-dependent strength between 1000°C and 1400°C. Optical examination of all bars showed that the carbon core of the SiC fibers was no longer present; there was oxidation of the matrix and, in many cases, a white foam was present around the cracks on the far ends of the bars (see Figures 6 and 7).

The loss of the fiber carbon core is due to oxidation. The silicon nitride matrix is also susceptible to oxidation after long-term exposure at elevated temperatures.[7-9] The white foam is probably due to burnoff of the excess binder. The ends of each fiber layer are coated with a large amount of binder to maintain the fiber spacing within the layer during processing of the CMC. Apparently, the processing conditions are not sufficient to burn off all the binder, thus it is burned off during elevated temperature testing leaving behind a white foam.*

*LECOSTAOUEC, J. F. Textron Specialty Materials, private communications.

7. CLARKE, D. R., and LANGE, F. F. *Oxidation of Si_3N_4 Alloys: Relation of Phase Equilibria in the System Si_3N_4-SiO_2-MgO*. J. Am. Ceram. Soc., v. 63, no. 9-10, 1980, p. 586-593.

8. QUACKENBUSH, C. L., and SMITH, J. T. *Phase Effects in Si_3N_4 Containing Y_2O_3 or CeO_2: II, Oxidation*. J. Am. Ceram. Soc., v. 59, no. 5, 1980, p. 533-537.

9. QUACKENBUSH, C. L., SMITH, J. T., and NEIL, J. *Oxidation in the Si_3N_4-Y_2O_3-SiO_2 System*. Proceedings of the 18th Automotive Technology Development Contractors' Coordination Meeting, Society of Automotive Engineers, Warrendale, PA, November 1980, p. 11-14.

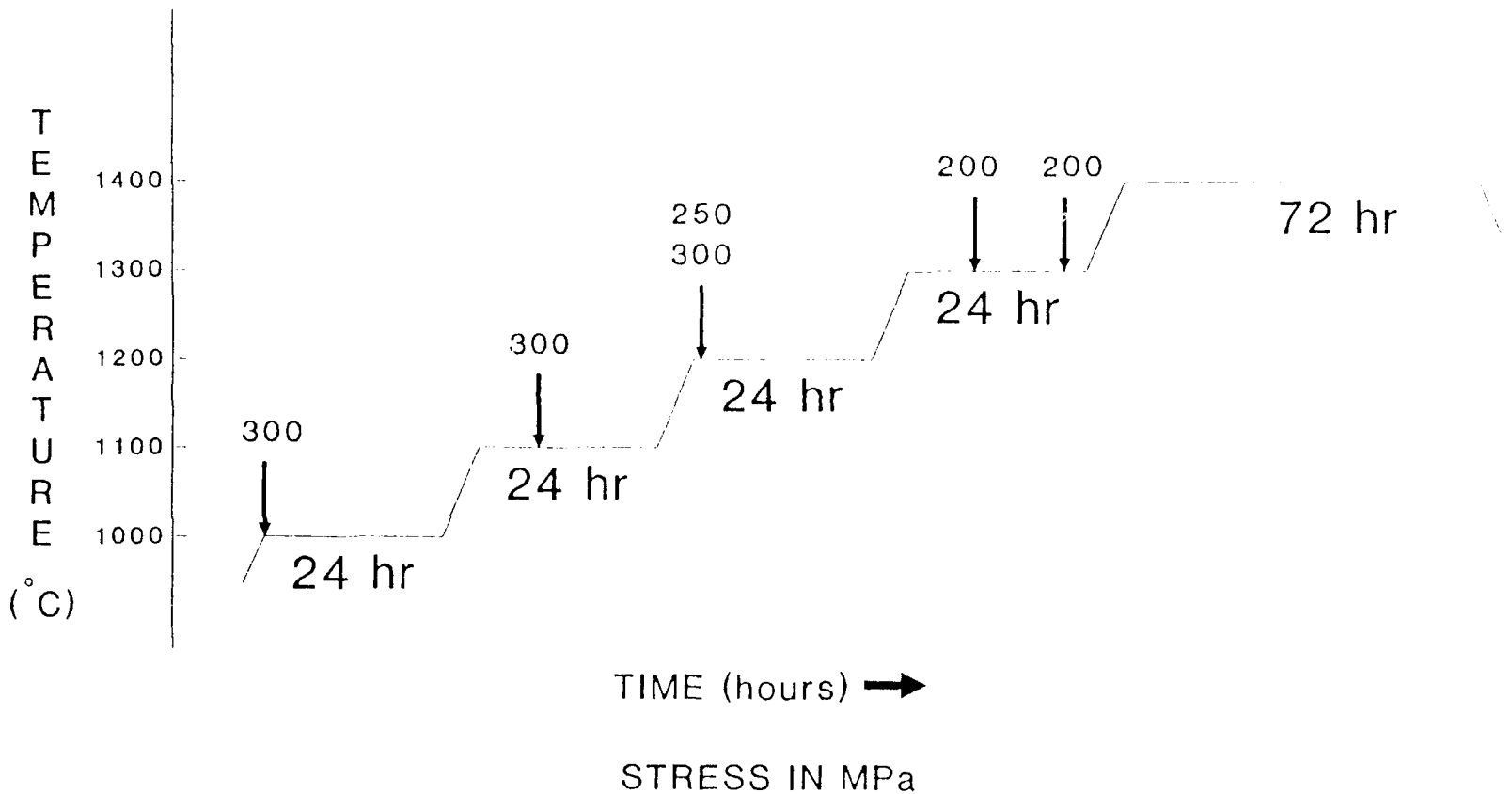


Figure 5. Stepped-temperature stress-rupture results for the Textron CMC.

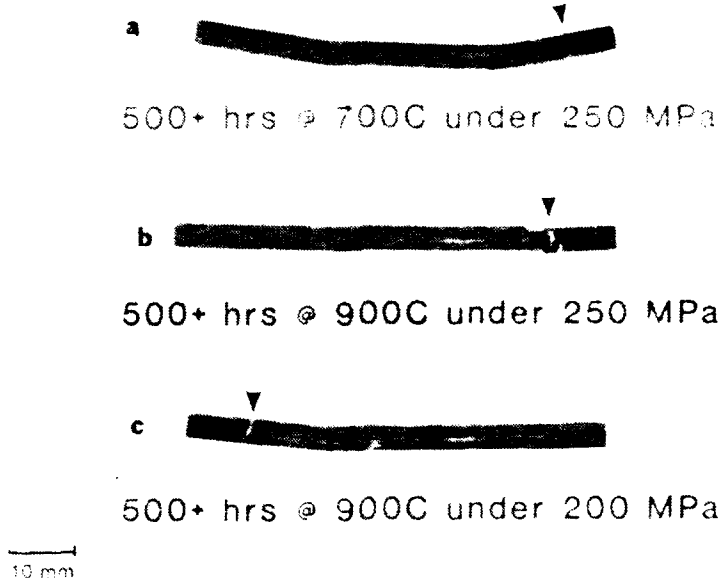


Figure 6. Failure at room temperature after long duration stress rupture testing. Bars a and c fractured at the load pins but were not tested to ultimate rupture. Black arrows show white foam.

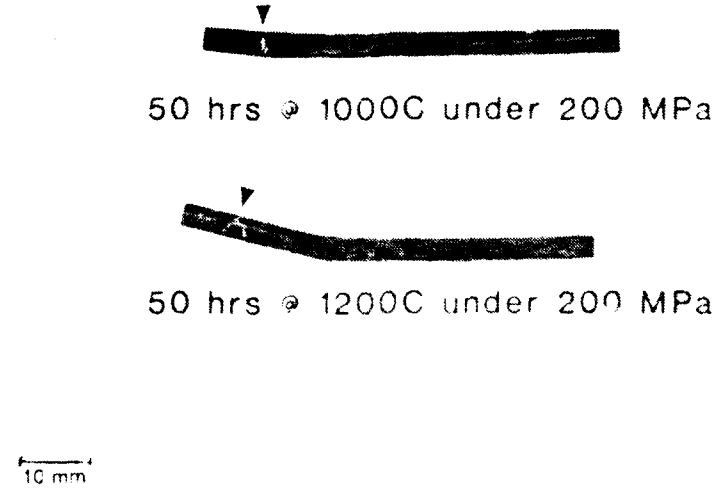


Figure 7. Failure at room temperature after 50 hours of stress rupture testing. Both bars fractured but were not tested to ultimate rupture. Black arrows show white foam.

In all cases failure appeared to initiate in a tensile manner and then changed to a shear failure halfway through the bar, approximately at the location of the neutral axis. This is similar to the type of failure seen by Mah, et al.[10] in a fiber-reinforced, glass-ceramic-matrix composite. Some failures initiated where fibers were, or had been exposed, on the surface or at channels left behind when the fibers were removed during machining. Some fiber pullout was evident in the specimen, but the number of fibers which pulled out was significantly lower than at room temperature. In addition, the average pullout length was about half as long as at room temperature. This reduction in both the number and length of pullout (related to the room temperature tests) shows that the strength of the fiber/matrix bond is increasing.

Stress Rupture

Isothermal SR tests were carried out at various temperatures (see Table 1). Long duration specimens were allowed to run until failure, or 500 hours had elapsed, while the shorter ones ran only for 50 hours. There was no evidence of any unusual degradation at either 700°C or 900°C. Three of the long duration tests; one at 700°C and two at 900°C, survived for 500+ hours under 250 MPa applied stress without failure but with a small amount of permanent creep deformation. The creep deformation could be traced to the matrix materials, the interface, or the fibers themselves, since CVD fibers manufactured from silanes are often nonstoichiometric and free Si may be present.[11,12]

Table 1. STRESS RUPTURE TEST MATRIX

Temperature (°C)	Stress (MPa)	Time (Hours)	Remarks
700	250	500	Survived
900	200	522	Survived
900	250	55	Failed
900	250	521	Survived
1000	200	50	Survived
1200	200	50	Survived
1200	200	58	Failed

As was the case with the bars used in the STSR tests, the carbon cores of the fiber were no longer present upon completion of the test; oxidation of the matrix was obvious and a white foamy substance was present near the ends of some of the bars. For the one bar that did not survive at 900°C, the failure mode and the amount of fiber pullout was the same as in the STSR tests, indicating an increase in the bond strength.

10. MAH, T., MENDIRATTA, M. G., KATZ, A. P., RUH, R., and MAZDIYASNI, K. S. *High-Temperature Mechanical Behavior of Fiber-Reinforced Glass-Ceramic Matrix Composites.* J. Am. Ceram. Soc., v. 68, no. 9, 1985, C-248-C-251.
11. BRUN, M. K., and BOROM, M. P. *Thermomechanical Properties of Chemically Vapor Deposited Silicon Carbide Filaments.* J. Am. Ceram. Soc., v. 72, no. 10, 1989, p. 1993-1996.
12. CHEN, L., GOTO, T., and HIRAI, T. *Preparation of Silicon Carbide Powders by Chemical Vapor Deposition of the SiH_4-CH_4-H_2 System.* J. Mat. Sci., 1989, p. 3824-3830.

The bar subjected to 1200°C under an applied stress of 200 MPa failed after ≈58 hours. The failure mode was brittle as there was no fiber pullout and the crack propagated in a perpendicular direction from the tensile surface through the entire thickness of the bar. This indicates that at this temperature there is a significant increase in the strength of the fiber/matrix interface which eliminates the likelihood of fiber pullout or crack deflection as a toughening mechanism.

At the request of Textron, two other SR tests were performed. Both were for 50 hours under a 200 MPa applied stress; one was at 1000°C and the other at 1200°C. The purpose of these tests was to determine the effect of temperature and stress on the room temperature behavior. Neither bar failed during the tests, but in each case there was a small amount of permanent creep strain evident. X-ray diffraction on the surface of the 1200°C specimen showed that in addition to α- and β-Si_3N_4, there were strong peaks for β-$Y_2Si_2O_7$ and α-SiO_2. A powdered bulk pattern was also run and did not show the latter two phases, but some minor unidentifiable broad peaks at d-spacings of 2.98, 2.79, 2.71, and 2.03 angstroms were seen.

Room Temperature Properties After Stress Rupture

The three bars which survived the long duration SR tests, and the two bars subjected to the short duration SR tests, were examined at room temperature using four-point flexure. None of the bars failed in a brittle manner (see Figures 6 and 7), yet the failure was not similar to that at room temperature without exposure. Three of the bars failed at or very near the load pin(s), while the other two failed outside the inner gauge length where the white foam appeared on the bar. There was no apparent trend in the failure mode between long and short duration SR tests.

Fractography

The fractography of the room temperature fracture surfaces showed fiber pullout and debonding with crack bridging as the toughening mechanism, as shown in Figure 4. The mechanisms are a direct result of debonding between the fiber and matrix. Because this material did not fail in a brittle manner, it was impossible to determine a failure origin. This was not unexpected.

Surprisingly, the analysis of the fracture surfaces from STSR tests revealed a fracture mirror and origin in the matrix, as shown in Figure 8. The way in which the crack propagated around the fibers provided a form of "hackle" which made it very easy to pinpoint the origin. Unlike the room temperature case, there is no evidence of debonding and the associated crack bridging. The amount and length of fiber pullout is also greatly reduced.

Fractographic analysis of the SR specimens also showed a clear failure origin. In one case, failure occurred due to a porous region ≈100 μm in size, as shown in Figure 9. The previous work here has shown that when the fibers are exposed on the surface, the failure will initiate in this area during room temperature testing. However, in this instance the failure originates in the matrix below a layer of exposed fiber even though the fibers are larger than the flaw, ≈125 μm verses ≈100 μm. This indicates that there is now a strong bond between the fiber and matrix, such that the material acts like a monolithic piece when the crack propagates through the specimen.

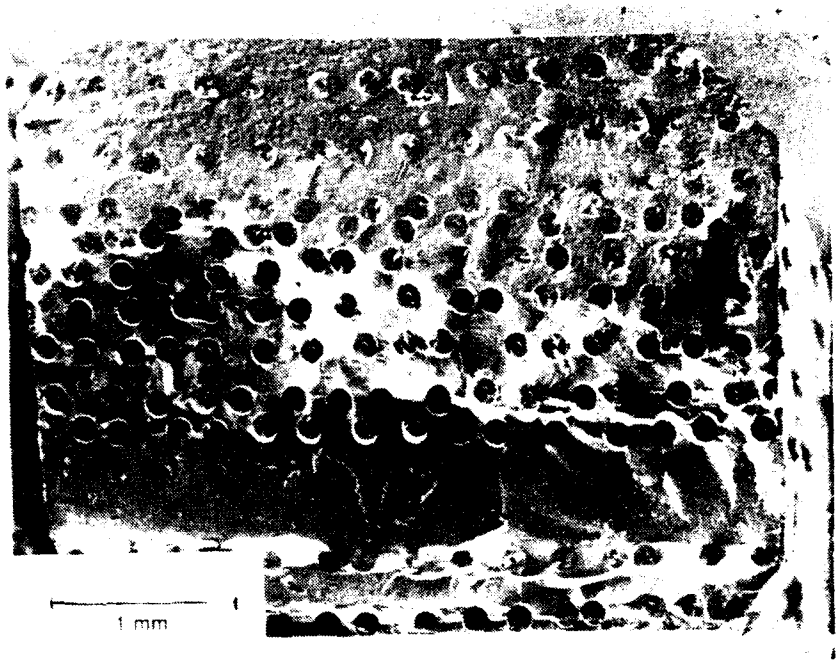


Figure 8a. Fracture surface from the STSR test. White arrow indicates failure origin. Tensile surface is at the top of the photo.

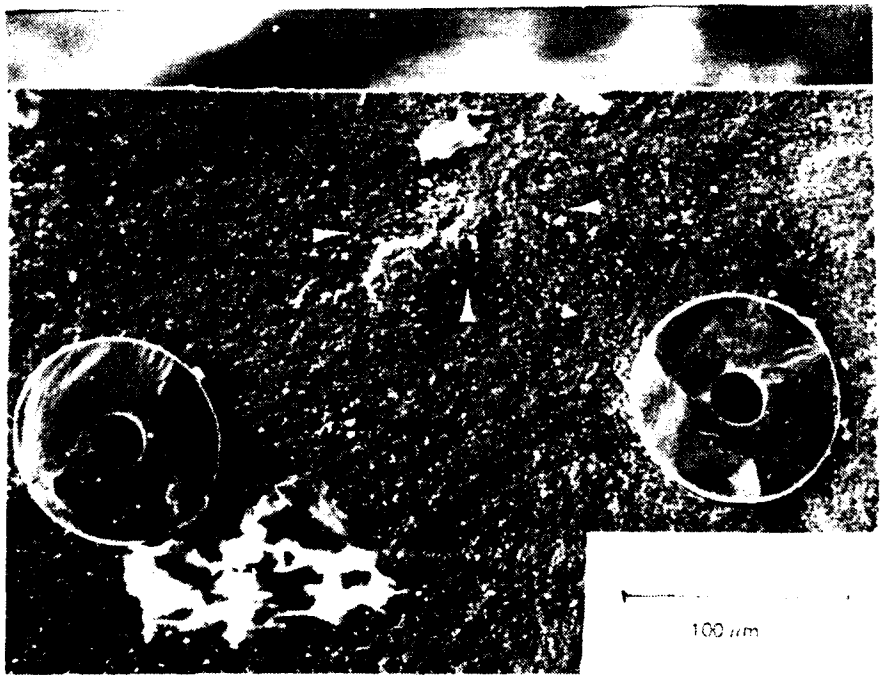


Figure 8b. Magnified photo of fracture origin. Origin (highlighted by arrows) appears to be a porous region. Tensile surface is at the top of the photo.

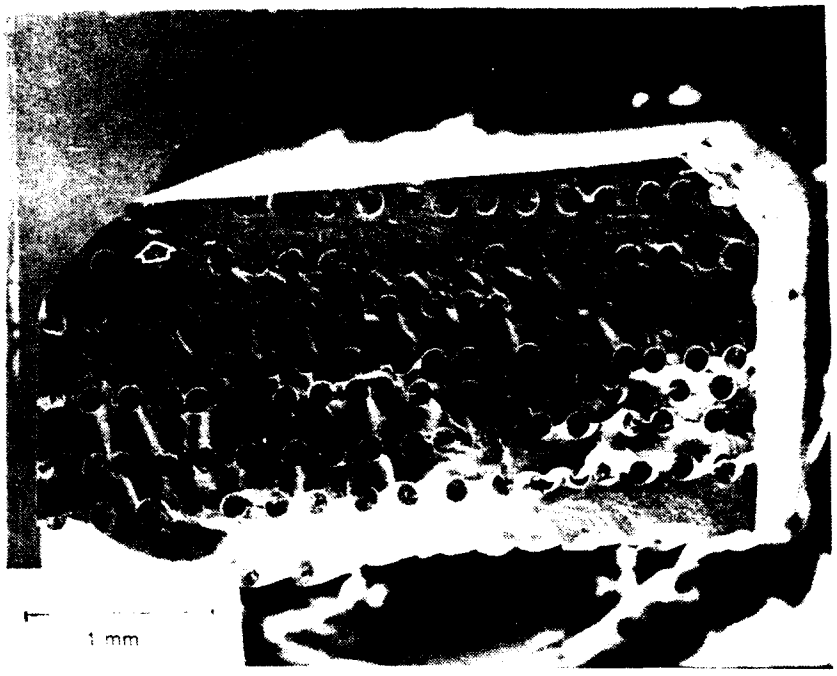


Figure 9a. Fracture surface from the SR test. Arrow indicates failure origin. Tensile surface is at the top of the photo.

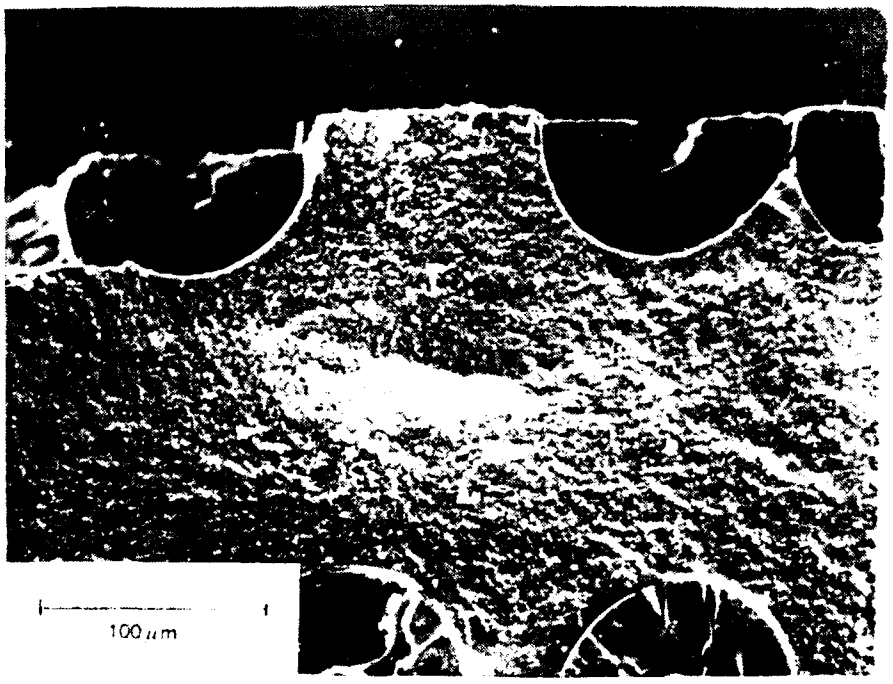


Figure 9b. Magnified photo of fracture origin. Origin (highlighted by arrows) appears to be a porous region. Tensile surface is at the top of the photo NOTE. The failure occurred in the matrix below some exposed fibers

SUMMARY

The preliminary work done in this report shows that continuous fibers can be used to increase the toughness of a ceramic material at room temperature. The addition of the fiber layers eliminates catastrophic brittle failure. As the test temperature increases, the tendency for failure to proceed in a brittle manner increases. This is most likely due to an increase in the fiber matrix bond strength which does not allow for fiber pullout and crack bridging to occur.

ACKNOWLEDGMENTS

The work described herein is part of the Characterization of Toughened Ceramics Program which is a subtask of the Department of Energy (DOE) sponsored, Oak Ridge National Laboratory (ORNL) monitored, Ceramic Technology for Advanced Heat Engines Project (Interagency Agreement DE-AI05-84OR21411). The purpose of this subtask is to examine commercial and experimental toughened ceramics for possible application in advanced heat engines.

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Technical Note MTL TN 90-2, September 1990, 16 pp - illus-table

Key Words

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Silicon nitride
Silicon carbon fiber

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